U S DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE ATTORNEY'S DOCKET NO FORM PCT 1390 HAJEK ET AL-1 (PCT) TRANSMITTAL LETTER TO THE UNITED STATES U.S. APPLICATION NO. (1f known, see 37 CFR 1.5) DESIGNATED/ELECTED OFFICE (DO/EO/US) 018119 CONCERNING A FILING UNDER 35 U.S.C. 371 INTERNATIONAL APPLICATION NO INTERNATIONAL FILING DATE PRIORITY DATE CLAIMED PCT/CZ00/00042 JUNE 12, 2000 JUNE 17, 1999 METHOD AND APPARATUS FOR HEAT TREATMENT OF GLASS MATERIAL AND TITLE OF INVENTION NATURAL MATERIALS SPECIFICALLY OF VOLCANIC ORIGIN APPLICANT(S) FOR DO/EO/US MILAN HAJEK ET AL. Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information: 1. X This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371. ___ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371. X This is an express request to begin national examination procedures (35 U.S.C. 371 (f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(l). X A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date. X A copy of the International Application as filed (35 U.S.C. 371(c)(2) (in English) a. X is transmitted herewith (required only if not transmitted by the International Bureau) b. ____ has been transmitted by the International Bureau. c. ____ is not required, as the application was filed in the United States Receiving Office (RO/US). A translation of the International Application into English (35 U.S.C. 371(c)(2)). Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)). a. ____ are transmitted herewith (required only if not transmitted by the International Bureau). b. ____ have been transmitted by the International Bureau. c. ____ have not been made; however, the time limit for making such amendments has **NOT** expired. d. ____ have not been made and will not be made. A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)). ___ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). 10. ___ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)). Items 11. to 16. below concern other document(s) or information included: 11. An Information Disclosure Statement under 37 CFR 1.97 and 1.98. 12. ___ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included. 13. X A FIRST preliminary amendment. ___ A SECOND or SUBSEQUENT preliminary amendment. 14. ___ A substitute specification. 15. ___ A change of power of attorney and/or address letter. 16. X Other items or information: 1 Sheet of Formal Drawings Applicant Claims Priority under 35 U.S.C. §119 of Czech Republic Application No. 1999-2185 filed June 17, 1999. Applicant Claims Priority under 35 U.S.C. §120 of: PCT No. PCT/CZ00/000042 filed June 12, 2000.

ii C CUC18 Recid PCT/PTO- ii 4 10 EC 2001

APPLICATION NO. (i.c. known, sec 37 GFR 15) 8 1 1 9				INTERNATIONAL APPLICATION NO PCT/CZ00/00042	ATTORNEY'S DOCKET NO HAJEK'ET AL-1 PCT			
_X The followin	X The following fees are submitted			CALCULATIONS	PTO USE ONLY			
Basic National Fee	Basic National Fee (37 CFR 1.492(a)(1)-(5)):							
Search Report has be	Search Report has been prepared by the EPO or JPO							
International preliminary examination fee paid to USPTO (37 CFR 1 482)								
international search	l preliminary examination fee (37 CFR 1 445(a)(2)) _I	oaid to USPTO\$1,04						
International prelimi and all claims satisfi	mary examination fee paid ed provisions of PCT Arti	to USPTO (37 CFR 1 48) cle 33(2)-(4). \$1	\$ 890 00					
	ENTER APPRO	PRIATE BASIC FEE A	. 030 00					
Surcharge of \$130.00 for months from the earlies	or furnishing the oath or do t claimed priority date (37	eclaration later than 2 CFR 1 492(e)).						
Claims	Number Filed	Number Extra	Rate					
Total Claims	10 - 20 =	- 0 -	X \$18.00	\$				
Independent Claims	1 - 3 =	- 0 -	X \$84.00	\$				
Multiple dependent	claım(s) (ıf applıcable)		+ \$280.00	\$				
	TOTAL OF	ABOVE CALCULATION)NS =	\$ 890 00				
Reduction by 1/2 for Sn	nall Entity status			\$ 445.00				
		SUBTOTAL =		\$ 445 00				
	00 for furnishing the Engli t claimed priority date (37		2030	\$				
200	Т	OTAL NATIONAL FEE	Σ =	\$ 445 00				
	closed assignment (37 CF ropriate cover sheet (37 CI			See cover sheet attached to assign \$ to be charged to Deposit Acet				
	7	OTAL FEES ENCLOSI	\$ 445.00					
			Amount to be	,				
				refunded	\$			
				charged	\$			
 X Applicant claims Small Entity status. a. X A check in the amount of \$445.00 to cover the above fees is enclosed. b. Please charge my Deposit Account No. 03-2468 in the amount of \$ to cover the above fees. A duplicate copy of this sheet is enclosed. c. X The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment, to Deposit Account No. 03-2468. A duplicate copy of this sheet is enclosed. 								
NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.								
SEND ALL CORR	ESPONDENCE TO:		01	. 1/4/				
COLLARD & ROE			ero tean	8r				
1077 Northern Bou			Signature					
Roslyn, New York	11576-1696							
(516) 365-9802			Edward R. Fre Reg. No. 26,04					
	Express Mail No. <u>EL 871 448 394 US</u>							
Date of Deposit December 14, 2001 I hereby certify that this paper or fee is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37								
CFR 1.10, on the date indicated above, and is addressed to BOX PCT, U.S. Patent and Trademark Office, P.O. Box 2327, Arlington, VA 22202.								
	<u>OMUL A - (MUPU)</u> Lisa L. Vulpis							

10019119 102018119 1

JC13 Rec'd PCT/PTO 1 4 DEC 2001

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANT:

MILAN HAJEK ET AL-1 (PCT)

PCT No.:

PCT/CZ00/00042

FILED:

JUNE 12, 2000

TITLE:

METHOD AND APPARATUS FOR HEAT TREATMENT OF GLASS MATERIAL AND NATURAL MATERIALS SPECIFICALLY OF

VOLCANIC ORIGIN

PRELIMINARY AMENDMENT

BOX PCT

U.S. Patent and Trademark Office P.O. Box 2327 Arlington, VA 22202

Dear Sir:

Preliminary to Examination, please amend the aboveidentified application as follows:

IN THE SPECIFICATION

Page 1, after the title, please insert as follows:

-- CROSS REFERENCE TO RELATED APPLICATIONS

Applicant claims priority under 35 U.S.C. §119 of Czech Republic Application Nos. PV 1999-2185, PV 2000-968, and PV 2000-1935 filed June 17, 1999, March 17, 2000, and May 25, 2000, respectively. Applicant also claims priority under 35 U.S.C. §120 of PCT/CZ00/00042 filed June 12, 2000. The international application under PCT article 21(2) was published in English.--

IN THE CLAIMS

Please amend claims 3, 5, 6, and 8-10 as follows:

- 3. (Amended) The method of heat treatment of glass and natural materials of claim 1 characterized in that the glass or natural material to be melted or refined and/or refined contains an inert additive elected from the group comprising carbides, nitrides or borides in an amount from 1 to 100 g preferably 5 to 50 g per 1 kg of the glass or natural material.
- 5. (Amended) The method of heat treatment glass and natural materials of claim 1 characterized in that the glass material comprises cullet of common waste glass of any kind or glass batches of all types or mixtures of cullet and glass and glass batches and the natural material comprises basalt, granite, marble, andesite, syenite, and other materials absorbing micro wave radiation.
- 6. (Amended) An apparatus for performing the method of claim 1 characterized in that it consists substantially of a micro wave furnace comprising an outer shell (8.2) provided with a cover (10) and an inner shell (8.1) and at least one micro wave generator (1.1, 1.2, 1.3, 1.4) with double emission and a total output from 0.1 to 1 kW per 1 kg of the processed glass or natural material arranged substantially in the intermediate space between the outer shell (8.2) and the inner shell (8.1) and a tank (2) disposed inside the inner shell (8.1).

- 8. (Amended) The apparatus of claim 6 characterized in that the furnace cover (10) is provided with at least one safety switch (9.1 and 9.2) and a fill neck (7) engaging a contactless infrared sensor (5) with a connection for transmitting its signal to a thermometer and controller (6) provided with a microprocessor for the microwave generator control.
- 9. (Amended) The apparatus of claim 6 characterized in that the tank (2) is provided with a side or bottom tapping point (13).
- 10. (Amended) Apparatus of claim 6 characterized in that the outer shell (8.2) is provided with transporting wheels.

A marked-up version is shown as Exhibit A.

Please add the <u>Abstract</u>, attached hereto on a separate sheet.

REMARKS

By this Preliminary Amendment, a cross-reference to related applications has been inserted in page 1. Claims 3, 5, 6, and 8-10 have been amended so that the multiple dependency of certain of the dependent claims have been removed to avoid the surcharge associated therewith, and an Abstract is being provided. No new

matter has been introduced. Entry of this amendment is respectfully requested.

Respectfully submitted,

MILAN HAJEK ET AL

By:

Allison C. Collard, Reg. No. 22,532 Edward R. Freedman, Reg. No. 26,048

Attorneys for Applicant

COLLARD & ROE, P.C. 1077 Northern Boulevard Roslyn, New York 11576 (516) 365-9802 ERF/llv

Enclosure:

Exhibit A and an Abstract

EXPRESS MAIL NO. EL871448394US
Date of Deposit: December 14, 2001

Date of Deposit: <u>December 14, 2001</u>
I hereby certify that this paper or fee is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10, on the date indicated above, and is addressed to BOX PCT, U.S. Patent and Trademark Office, P.O. Box 2327, Arlington, VA 22202.

Lisa L. Vulpis

ABSTRACT OF THE DISCLOSURE

A method of heat treatment of glass materials and natural materials specifically of volcanic origin according to which the treated material is exposed to microwave radiation at a frequency range from 1 MHz to 10 GHz and temperature range from the ambient temperature to 1800°C in a batch or continuous process. The glass or natural material subjected to a melting and/or refining process contains an inert additive elected from the group comprising carbides, nitrides or borides in an amount from 1 to 100 g preferably 5 to 50 g per 1 kg of the glass or natural material. The apparatus consists substantially of a microwave furnace comprising an outer shell (8.2) provided with a cover (10) and an inner shell (8.1) and at least one micro wave generator (1.1, 1.2, 1.3, 1.4) with double emission and total power from 0.1 to 1 kW per 1 kg of the treated material.

3

EXHIBIT A

VERSION WITH MARKINGS TO SHOW CHANGES MADE TO CLAIMS 3, 5, 6, AND 8-10

- 3. (Amended) The method of heat treatment of glass and natural materials of claim 1 [and 2] characterized in that the glass or natural material to be melted or refined and/or refined contains an inert additive elected from the group comprising carbides, nitrides or borides in an amount from 1 to 100 g preferably 5 to 50 g per 1 kg of the glass or natural material.
- 5. (Amended) The method of heat treatment glass and natural materials of [any of claims 1 to 4] claim 1 characterized in that the glass material comprises cullet of common waste glass of any kind or glass batches of all types or mixtures of cullet and glass and glass batches and the natural material comprises basalt, granite, marble, andesite, syenite, and other materials absorbing micro wave radiation.
- 6. (Amended) An apparatus for performing the method of [any claim 1 to 5] claim 1 characterized in that it consists substantially of a micro wave furnace comprising an outer shell (8.2) provided with a cover (10) and an inner shell (8.1) and at least one micro wave generator (1.1, 1.2, 1.3, 1.4) with double emission and a total output from 0.1 to 1 kW per 1 kg of the processed glass or natural material arranged substantially in the intermediate space between the outer shell (8.2) and the inner shell (8.1) and a tank (2) disposed inside the inner shell (8.1).
- 8. (Amended) The apparatus of claim 6 [or 7] characterized in that the furnace cover (10) is provided with at least one safety switch (9.1 and 9.2) and a fill neck (7) engaging a contactless infrared sensor (5) with a connection for transmitting its signal to a thermometer and controller (6) provided with a microprocessor for the microwave generator control.
- 9. (Amended) The apparatus of [any claim 6 to 8] claim 6 characterized in that the tank (2) is provided with a side or bottom tapping point (13).
- 10. (Amended) Apparatus of [any claim 6 to 9] claim 6 characterized in that the outer shell (8.2) is provided with transporting wheels.

WO 00/78684

1 pats

JC13 Rec'd PCT/PTO 1 4 DEC 2001 PCT/CZ00/00042

Method and apparatus for heat treatment of glass materials and natural materials specifically of volcanic origin

Field of invention

The invention relates to a method of heat treatment glass and natural materials specifically of volcanic origin. Under the heat treatment of glass materials it is to be understood melting or refining of glass cullet, glass batches or mixtures thereof or hardening or forming glass. The materials of volcanic origin such as basalt, granite, marble, andesite, syenite, etc. are accordingly subjected to melting or refining or hardening and forming to obtain utility goods such as floor tiles, wall tiles, rods, bars, fibers, insulating wool, artistic objects and various glassware etc. In addition, the invention relates to an apparatus for performing this method.

Description of prior art

At the present time the melting of glass or production of glass melt or mother glass as well as melting of natural materials specifically of volcanic origin such as basalt is effected almost exclusively in glassmaking furnaces or melting furnaces heated by gas burners. Such furnaces show heavy weight and robustness. The furnace is to be heated as a whole and must be provided with thick insulation layers - fireclay what considerably affects its mobility i.e. the possibility of moving it from place to place for example for exhibition purposes. Moreover, the combustion of considerable amount of gas results in the generation of notably amount of hazardous flue gases affecting environment and also in strong heat radiation into the working place area what makes the working conditions unpleasant. There have been efforts to replace such glassmaking furnaces by electrically heated furnaces but due to specific parameter requirements such as temperature, glass output and energy consumption such efforts resulted in their limited use mostly for economic reasons. The requirement for quick melting of glass or natural materials by classic heating methods faces the main problem of considerably low thermal conductivity of such materials. In addition, certain types of glasses or materials especially those containing iron such as welding glass or basalt effectively reflect the infrared radiation so the temperature profile is sharply decreasing from the surface to the inside of the material so that the thickness of the batch material must be limited.

Attempts to make use of a micro wave technology has encountered serious difficulties since most types of glass are well transparent for micro wave i.e. glass

2

does not absorb microwaves at the ambient temperature and has to be activated in some way i.e. make it capable to absorb microwaves. It is also known that at certain temperature - about 500 °C and more the positively charged particles of alkaline ions vibrating in the negative charged interstitial position begins to act as oscillating dipole which is the basic condition for absorption of microwaves. The pre-heating may be effected for example by electrical heating what of course requires a hybrid furnace i.e. a furnace provided with combined electric and microwave heating which design is relatively costly and restrictive in capacity. Some authors have used for the preheating of transparent materials such as asbestos and kieselguhr various additives capable to absorb microwaves, for example powdered iron, iron trichloride, or borax (F.G. Wihsmann, R. Kokoschko, K. Forkel, "Glassmaker and Ceramicmaker" 46 75 (1996). However, such materials proved to be unsuitable as additives to glass materials since they react with the glass mass and undesirably change the glass composition and structure. Other authors used for the batch preheating a microwave absorbing envelop or they hydrated the material before its melting (M.P. Knox, Gl.J. Copley, "Glass technology" 38, 91 (1997). Neither these activating methods are perfect since by using a wave absorbing envelop the microwaves are hindered from penetrating into the batch and the heating is distributed by radiation as in the case of the classic heating processes. On the other hand with the majority of types of glass neither the hydration nor wetting provides sufficient means to warm up the batch to the temperature required.

According to EP-A1-0 349 405 micro waves are used for preheating or heating of materials such as oxides, glass and certain metals producing corrosive effect on the furnace lining when subjected to an induction heating. To enable preheating of materials that are not susceptible to micro waves glass containing iron oxides is added to the batch which oxide due to its high micro wave absorption provides for heating of the remaining micro-waves not susceptible materials. Nevertheless, such process is unacceptable for production of most of types of glass or natural materials based products since the iron oxide substantially affect the required qualities of final products.

The object of the invention is to provide a new method of heat treatment of glass materials and natural materials specifically of volcanic origin which enables melting, refining or hardening of such materials under specific conditions by applying the microwave technology in the full temperature range required and to all types of materials notwithstanding the composition or structure thereof.

R1.34

2a

Another object of the invention is to provide an apparatus for performing said method operated in both the batch or continuous process.

Summary of the invention

In accordance with the foregoing the treated material is exposed to microwave radiation at frequency range from 1 MHz to 10 GHz and temperature range from the ambient temperature to 1800 °C in a batch or continuous production process.

More specifically, the frequency of the microwave radiation is elected within the range from 1 to 100 MHz, preferably 27 MHz, from 500 MHz to 10 GHz preferably 896 MHz, 915 MHz and 2450 MHz.

To ensure safe and quick heating throughout the full temperature range the glass or natural material that is subjected to a melting and /or refining process contains an inert additive selected from the group comprising carbides, nitrides or borides in an amount from 1 to 100 g preferably 5 to 50 g per 1 kg of the glass or natural material.

More specifically, the inert additive is elected from the group consisting of tungsten carbide - WC, silicon carbide - SiC, boron carbide - B₄C, titanium carbide - TiC or vanadium nitride - VN, boron nitride - BN, silicon nitride - Si₃N₄ or titanium boride - TiB₂, niobium boride - NB₂, vanadium boride - VB₂, tungsten boride - WB₂, zirkonium boride ZrB₂, and aluminum boride AlB₂ or a mixture thereof.

The glass material may comprise glass cullet form common waste glass of any kind or glass batches of all types or mixtures of glass cullet and glass batch and the natural material may comprise basalt, granite, marble, andesite, syenite, and other materials absorbing the micro wave radiation.

An apparatus for performing the method consists substantially of a microwave furnace comprising an outer shell provided with a cover and an inner shell and at least one microwave generator with double emission and total power from 0.1 to 1 kW per 1 kg of the processed glass or natural material arranged substantially in the intermediate space in between the outer shell and inner shell and a tank disposed inside the inner shell.

The inner space of the furnace is advantageously filled up with a heat insulating material having a heat resistance up to 1750 °C elected from the group consisting of aluminum oxide - corrundum and silicon oxide - quartz and the furnace cover is provided with at least one safety switch and a fill neck engaging a contactless infrared sensor with a connection for transmitting its signal to a thermometer and controller provided with a microprocessor for the microwave generator control. To enable continuous process the tank is provided with a side or bottom tapping point. The easy mobility of the furnace is secured by transporting wheels mounted on the outer shell.

The method and apparatus according to the invention are based on the application of microwave energy for selective heating of glass, glass materials, natural materials especially of volcanic origin such as basalt, granite, marble etc.. The applied technology may ensure that only the material required to be heated up is exposed to the heating effect uniformly in its whole volume while the adjacent space remains unaffected by the heat. In this way the supplied energy is used exclusively for

melting, refining or hardening of the material required and it is not necessary to heat up the whole body of the furnace.

Another advantage of the invention results from applying inert materials (for example silicone carbide) as additives to the glass mass or batch. Such inert materials are strong absorbers of microwaves even at the ambient temperature—while the properties of glass or natural materials remain unaffected. In this way any type of glass may be melted notwithstanding the extent to which the glass is capable to absorb microwaves as well as the glass composition and particles size including any glass batches or natural materials specifically of volcanic origin containing a metal for example. The melting process is extremely accelerated and is determined only by the heat resistance of a ceramic crucible. A metallic or graphite crucible cannot be used due to their unfavorable interaction with microwaves.

Any undesirable phenomena such as material loss or its oxidation by air oxygen are fully suppressed in the microwave melting process. The required properties of the material processed are completely preserved yet may be altered by a controlled modification of the melting regime. For example, by an appropriate application of the microwave energy in a glass batch melting process glass of different properties may be obtained that cannot be produced in the classic glassmaking furnaces (for example with respect to its morphology, microstructure or mechanical strength etc.).

The advantages of the invention based on the application of microwave heating technology including modified microwave furnace may be summarized as follows:

quick and in volume heating - the in volume heating means the effect of microwaves to heat materials almost uniformly from the volume center to its outer border in contrast to the classic heating;

selective heating - the selective heating means that only the material required is heated up while its surrounding remains cool;

the furnace need not be permanently supplied by energy - the furnace may be switched on or off at any time, i.e. it need not be operated continuously;

low electric energy consumption resulting in substantially lower operation costs - this benefit arises from the preceding attributes;

harmless working conditions - no hazardous combustion gases are generated neither the temperature at the working place is increased.

In addition, besides for the mere melting purposes the furnace may be used for refining, hardening or forming of various glass materials, melting of several glass samples for example to serve the purpose of color decorations or processing of molten natural materials to produce utility goods such as floor tiles, wall tiles, rods, bars, fibers insulating wool artistic objects etc.

Brief description of drawings

The accompanied drawings shows o schematic vertical sectional view of one possible embodiment of the apparatus according to the invention.

Description of the preferred embodiment

Example 1

5 kg of crushed transparent glass cullet of particle size from 2 to 6 mm and 100 g of compact tungsten carbide (WC) were charged into a ceramic crucible with a capacity of 4 liters (1) in volume whereupon the crucible was put into a microwave furnace. After closing the furnace cover the crucible contents was heated up by means of microwave radiation with a frequency 2450 MHz and power 4 kW until the batch was melt. The glass melt was maintained at a temperature of 1200 \pm 50 °C and processed in forming various utility items.

Example 2

2 kg of the mixture consisting of a lead crystal batch and 50 g of compact tungsten carbide (WC) were charged into a ceramic crucible with the capacity of 4 liters then the crucible was put into a microwave furnace. After closing the furnace cover the crucible contents was heated by microwave radiation at a frequency of 2450 MHz and power 2 kW until the glass was melted and then the glass melt was refined at a temperature of 1450 °C and thereafter at 1200 ± 20 °C. The glass melt was further maintained at this temperature and utilized in production of various utility items.

Example 3

The glassmaking process according to example 2 was repeated under substantially the same conditions with the exception that as additives the following compounds were employed one after other: tungsten carbide -WC, silicon carbide -SiC, boron carbide - B₄C, titanium carbide - TiC or vanadium nitride - VN, boron

nitride - BN, silicon nitride - Si_3N_4 or titanium boride - TiB_2 , niobium boride - NB_2 , vanadium boride - VB_2 , tungsten boride - WB_2 , zirkonium boride ZrB_2 , and aluminum boride AlB_2 .

Example 4

10 kg of glass cullet originating from the waste packing glass such as bottles, jars etc. and 200 g of the compact tungsten carbide (WC) were placed inter a ceramic tank 10 liters by volume provided with a side or bottom tapping point. The tank was placed into a microwave furnace which was closed and switched on to run at the highest power. The glass cullet was melted and refined by the micro wave radiation effect and the glass melt was withdrawn through the bottom or a side tapping point to be further processed. The furnace was provided with inlet and outlet means so that the whole process could be carried out in a continuous mode.

Example 5

5 kg of glass crushed basalt of particle size from 0.2 to 60 mm were put into a ceramic crucible with the capacity of 4 liters and the crucible was placed into a microwave furnace. After closing the furnace the batch in crucible was heated by a micro wave radiation at a frequency of 2450 MHz and power 4 kW until the batch was completely melted at a temperature of 1600 °C and then this temperature was reduced to 1200 °C. The molten basalt was then maintained at 1200 \pm 20 °C and further processed in various utility goods.

Example 6

8 kg of crushed basalt of particle size from 0.2 to 60 mm were put into a ceramic crucible having the capacity of 10 liters and the crucible was put into a microwave furnace. After closing the furnace the batch in the crucible was heated by microwave radiation at a frequency of 915 MHz until the batch was completely molten at a temperature 1400 °C and then this temperature was decreased to 1200 °C. The molten basalt was then maintained at a temperature of 1200 °C and further formed by drawing to fibers or blowing to an insulating wool.

Example 7

30 kg of natural material selected from the group consisting of basalt, granite, marble, optionally in mixture with additives selected from the group of carbides,

nitrides and borides in an amount of 1 to 10 % by weight for the purpose of accelerating of the melting were put into a ceramic tank with the capacity of 20 liters. The material was melted by the effect of microwave energy and maintained molten at a temperature from 1400 to 1450 °C and then withdrawn through a bottom outlet. Simultaneously, the amount of the withdrawn molten material was compensated by substantially continuous supply of a raw material wherein the feed rate was controlled in order to maintain a substantially constant volume of the molten material in the tank.

Example 8

A batch or alternatively continuous operated glassmaking furnace comprises an outer shell 8.2 and an inner shell 8.1. The inner shell 8.1 defines a heat insulated inner space which is filled up with an insulating refractory material 3 of aluminum oxide - corundum. This material is permeable for microwaves even at high temperatures. Microwave generators called magnetrons 1.1, 1.2, 1.3, 1.4 are mounted on the inner shell 8.1 and extend into the intermediate space between the inner shell 8.1 and the outer shell 8.2. In this intermediate space fans 4 for cooling magnetrons 1.1 - 1.4. are located. The upper part of the glass furnace is provided with a cover 10 having an upwardly projecting fill neck 7. The fill neck 7 is connected over a conduit 12 to a storage reservoir 11 of batch material. The cover 10 is further provided by two safety switches 9.1, 9.2. The fill neck 7 is engaged with an infrared sensor 5 connected to a thermometer and temperature controller 6 equipped with a microprocessor for controlling the operation of the furnace. The bottom of the outer shell 8.2 is fitted with transport wheels 14. A tank 2 for receiving the batch material is situated in the insulated space its upper part being connected to the fill neck 7 while the bottom thereof is connected to the tapping point 13.

At least four micro waves generators - magnetrons 1.1 - 1.4 are installed to generate microwaves energy at a frequency of 2450 MHz. with single or double emission in order to provide as much as possible homogenous electromagnetic field. The total microwave power may be adjusted with respect to the quantity of the natural material batch within the range from 2 to 6 kW, preferably 4 kW per 10 to 15 kg of the batch. The temperature of the molten material is measured by a contactless infrared sensor 5 and regulated by a thermometer coupled to a controller 6 equipped with a process controlling microprocessor. The mechanical safety switches 9.1 and 9.2 provided on the cover 10 prevent the microwave radiation from scattering into

the furnace surroundings when the furnace is opened so that they switch off the energy supply to the magnetrons 1.1 - 1.4.

In operation, the batch material is supplied continuously or semi-continuously from the storage reservoir 11 to the tank 2 through the fill neck 12 where the batch material is melted and refined and subsequently withdrawn continuously or semi-continuously through the tapping point 13.

Industrial applicability

The invention may be used for melting or production of all types of glass and natural materials especially of volcanic origin notwithstanding the extent to which such materials are capable to absorb microwaves. The processes under invention in combination with a microwave furnace may be used in glass factories for laboratory purposes (for example for the preparation of common, modified or new types of glasses) for artistic purposes (production of artistic objects , replicas etc.) for decorative purposes (decoration of basic shapes with various kinds of colored glass).

In summary, the invention may be used in glass factories, laboratories, studious, artistic studious, home glass shops and similar works and in similar facilities for melting and processing basalt and like materials to produce insulating wool, fibers or utility items such as floor and wall tiles including without limitation vases, bowls and statues. Thanks to the easy mobility of the microwave furnace the inventive process and furnace may be used at exhibitions and fairs for demonstration of the production glassware and other goods of natural materials as a part of manufacturers promotion of their products as well as for teaching and training purposes at professional schools of applied and decorative arts.

Printed:20-08-2001

9

CLAIMS

- 1. A method of heat treatment of glass materials and natural materials specifically of volcanic origin characterized in that the treated material is exposed to microwave radiation at a frequency range from 1 MHz to 10 GHz and temperature range from the ambient temperature to 1800 °C in a batch or continuous production process in the presence of an inert additive elected from the group comprising carbides, nitrides or borides in an amount from 1 to 100 g.
- 2. The method of heat treatment of glass and natural materials of claim 1 characterized in that the frequency of micro wave radiation is ranging from 1 to 100 MHz, or from 500 MHz to 10 GHz.
- 3. The method of heat treatment of glass and natural materials of claim 1 and 2 characterized in that the frequency of micro wave radiation is 27 MHz or 896 MHz, or 915 MHz or 2450 MHz and the amount of the inert additive is from 5 to 50 g per 1 kg of the glass or natural material.
- 4. The method of heat treatment of glass and natural materials of claim 3 characterized in that the inert additive is elected from the group consisting of tungsten carbide -WC, silicon carbide SiC, boron carbide B₄C, titanium carbide TiC or vanadium nitride VN, boron nitride BN, silicon nitride Si₃N₄ or titanium boride TiB₂, niobium boride NB₂, vanadium boride VB₂, tungsten boride WB₂, zirkonium boride ZrB₂, and aluminum boride AlB₂ or a mixture thereof.
- 5. The method of heat treatment glass and natural materials of any of claims 1 to 4 characterized in that the glass material comprises cullet of common waste glass of any kind or glass batches of all types or mixtures of cullet and glass and glass batches and the natural material comprises basalt, granite, marble, andesite, syenite, and other materials absorbing micro wave radiation.

Printed:29-03-2001

P(+, 34

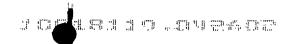
(...

6. An apparatus for performing the method of any claim 1 to 5 characterized in that it consists substantially of a micro wave furnace comprising an outer shell (8.2) provided with a cover (10) and an inner shell (8.1) and at least one micro wave generator (1.1, 1.2, 1.3, 1.4) with double emission and a total output from 0.1 to 1 kW per 1 kg of the processed glass or natural material arranged substantially in the intermediate space between the outer shell (8.2) and the inner shell (8.1) and a tank (2) disposed inside the inner shell (8.1).

10

- 7. The apparatus of claim 6 characterized in that the inner space of the furnace is filled up with a heat insulating material with a heat resistance up to 1750 °C selected from the group consisting of aluminum oxide - corundum or silicon oxide - quartz.
- 8. The apparatus of claim 6 or 7 characterized in that the furnace cover (10) comprises a fill neck (7), at least one safety switch (9.1 and 9.2) and further a contactless infrared sensor (5) is provided for sensing the temperature of the treated material through the fill neck (10) with a connection for transmitting the infrared sensor (5) signal to a temperature controller (6) provided with a microprocessor for the microwave generator control.
- 9. The apparatus of any claim 6 to 8 characterized in that the tank (2) is provided with a side or bottom tapping point (13).
- 10. Apparatus of any claim 6 to 9 characterized in that the outer shell (8.2) is provided with transporting wheels.





(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization International Bureau



(43) International Publication Date 28 December 2000 (28.12.2000)

PCT

(10) International Publication Number WO 00/78684 A1

(51) International Patent Classification7: C03C 1/00

C03B 5/02,

(21) International Application Number: PCT/CZ00/00042

(22) International Filing Date: 12 June 2000 (12.06.2000)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data: PV 1999-2185 17 June 1999 (17.06.1999) CZ PV 2000-968 17 March 2000 (17.03.2000) CZ PV 2000-1935 25 May 2000 (25.05.2000)

(71) Applicant (for all designated States except US): ÚS-TAV CHEMICKÝCH PROCESŮ AKADEMIE VĚD CESKÉ REPUBLIKY [CZ/CZ]; Rozvojová 135, 165 02 Praha 6 - Suchdol (CZ).

(72) Inventors; and

(75) Inventors/Applicants (for US only): HÁJEK, Milan [CZ/CZ]; Jugoslávských partyzánů 17, 160 00 Praha 6 (CZ). DRAHOŠ, Jiři [CZ/CZ]; Na zlatnici 22, 147 00 Praha 4 (CZ). VOLF, Václav [CZ/CZ]; Devotyho 179, 530 02 Pardubice (CZ). VOSÁB, Jaroslav [CZ/CZ]; 533 42 Živanice 39 (CZ).

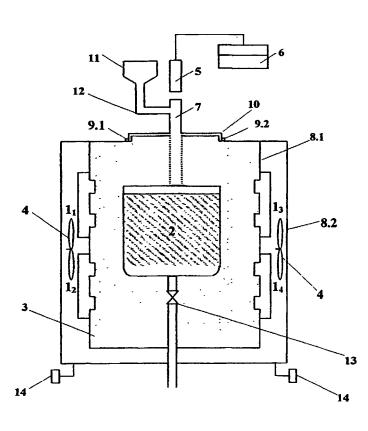
(74) Agent: REZÁC, Petr; Severovychodní-VI 629/9, 141 00 Praha 4 (CZ).

(81) Designated States (national): BR, CA, CN, ID, IL, IN, IS, JP, KP, KR, MX, NO, NZ, PL, RU, SG, SI, SK, TR, UA, US, VN, ZA.

(84) Designated States (regional): European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

[Continued on next page]

(54) Title: METHOD AND APPARATUS FOR HEAT TREATMENT OF GLASS MATERIALS AND NATURAL MATERIALS SPECIFICALLY OF VOLCANIC ORIGIN



(57) Abstract: A method of heat treatment of glass materials and natural materials specifically of volcanic origin according to which the treated material is exposed to microwave radiation at a frequency range from 1 MHz to 10 GHz and temperature range from the ambient temperature to 1800 °C in a batch or continuous process. The glass or natural material subjected to a melting and/or refining process contains an inert additive elected from the group comprising carbides, nitrides or borides in an amount from 1 to 100 g preferably 5 to 50 g per 1 kg of the glass or natural material. The apparatus consists substantially of a microwave furnace comprising an outer shell (8.2) provided with a cover (10) and an inner shell (8.1) and at least one micro wave generator (1.1, 1.2, 1.3, 1.4) with double emission and total power from 0.1 to 1 kW per 1 kg of the treated material.



WO 00/78684

0

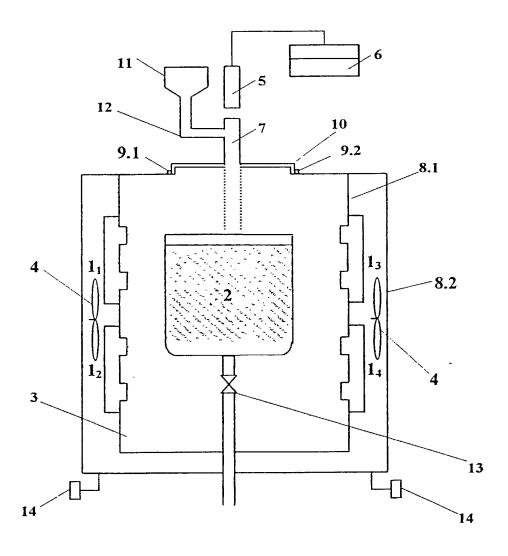


FIG. 1

COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (Includes Reference to PCT International Applications)

ATTORNEY'S DOCKET NUMBER HAJEK ET AL -1 (PCT)

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

METHOD AND APPARATUS FOR HEAT TREATMENT OF GLASS MATERIALS AND NATURAL MATERIALS SPECIFICALLY OF VOLCANIC ORIGIN

the specificatio	n of which (check only one item below):		
[]	is attached hereto.		
[]	was filed as United States application		
	Serial No.		
	on		
	and was amended		
	on	(if applicable).	
[x]	was filed as PCT international application		
	Number PCT/CZ00/00042		
	onJune 12, 2000		
a .	and was amended under PCT Article 19		
	on	(if applicable).	

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

PRIOR FOREIGN/PCT APPL	CATION(S) AND ANY PRIORI'	TY CLAIMS UNDER 35 U.S.C. 119:	

COUNTRY (if PCT, indicate "PCT")	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 35 USC 119
CZECH REPUBLIC	PV 1999-2185	17 JUNE 1999	[X]YES []NO
CZECH REPUBLIC	PV 2000-968	17 MARCH 2000	[X] YES [] NO
CZECH REPUBLIC	PV 2000-1935	25 MAY 2000	[X YES [] NO
		;	

	ereby claim the ber low.	nefit under Title 35, Unit	ed States Code	, Section i	19(e) of any Unite	d States pro	yvisional ap	plication(s) list
And by t	terion that is/are listed belo the first paragraph of Titl	Number) ider Title 35, United States Code owand, insofar as the subject matter to 35, United States Code, §112, ween the filing date of the prior	er of each of the cla Lacknowledge the	ed States appl tims of this app duty to discip	plication is not disclose in use material information :	that/those prior	rapplication(s)	in the mariner of evi
PR	IOR U.S. APPLICATIO	ns or pet internationa	L APPLICATION	s designa	ting the U.S. Por B		**************************************	120:
U.S. APPLICATIONS U.S. APPLICATION NUMBER U.S. RILING L'ATE					STATUS (
0	S. APPLICATION NUMBER	U.S. REAL	NO DATE		PATENTED	PBNI	DINO	ARANDONED
	, pa	T APPLICATIONS DESIGNATING THE			**************************************	ļ		
	PCT APPLICATION NO.	PCT PILING DATE	U.S. SERIAL NI	UMBERS (Facy)	,			<u> </u>
and AL ED	l Trademark Office conne LLISON C. COLLA DWARD R. FREED	As named inventor, I hereby appointed therewith. (List name and to IRD, Registration No. 22) MAN, Registration No. 18D RICHTER, Registration	registration number ,532; 26,048;	15): 1 F	KURT KELMAN, TROFRICK I. DO VILLIAM C. COL ROBERT W. GRIF	Registration RCH AK, F LARD, Reg	n No. <u>18,62</u> Rogistration Sistration No	8 No. 29,298 o. 38,411
Sei	nd Correspondence	to: COLLARD & ROE, 1077 Northern Boul Roslyn, New York I	evard	1 ,	Customer No. 2588	9		nhone Calls (o: elsphone m.inber) -9802
2	FULL NAME OF INVENTOR	PAMILY NAME HÁJEK	· · ,	PIRST GIVE			SECOND GIVE	in Pame
0	residence a Citizenship	erry 160 00 <u>Praha 6</u>			FOREIGN COUNTRY	CZX	CZECH!	CITIZENSHIP REPUBLIC
1	POST OFFICE ADDRESS	POST OPPICE ADDRESS Jugoslávských partyzá		160 00	Praha 6			CODE/COUNTRY REPUBLIC
			J 1 11				· · · · · · · · · · · · · · · · · · ·	
2	FULL NAME () OF INVEHTOR	DRAHOŠ		FIRST GIVE	NNAME.	-	SECOND GIVE	IN NAME
).		DRAHOS CITY 147 00 Praha 4	E	Jiři STATE OR É	OREION COUNTRY		COUNTRY OF	
)	residence &	DRAHOS city		STATE OR E CZECH	GREIGN COUNTRY		COUNTRY OF CZECH 1	CIMZENSHIP REPUBLIC
O. 2	OF INVEHTOR RESIDENCE & CITIZENSHIP POST OPPICE	DRAHOS CITY 147 00 PTABA 4 POST OFFICE ADDRESS		STATE OR E CZECH	Praha 4	22X	COUNTRY OF CZECH 1	CITIZENSHIP REPUBLIC CODE/COUNTRY REPUBLIC
2 2 2	OF INVENTOR RESIDENCE & CITIZENSHIP POST OFFICE ADDRESS FULL NAME	DRAHOS CITY 147 00 Praha 4 POST OFFICE ADDRESS Na zlatnici 22 FAMILY NAME		JIH STATE OR S CZECH CITY 147 00 MIRST CIVE VÁCIAV STATE OR F	Praha 4		COUNTRY OF CZECH I	CITIZENSHIP REPUBLIC ODE/COUNTRY REPUBLIC ON NAME
0 2	OF INVENTOR RESIDENCE & CITIZENSHIP POST OFFICE ADDRESS PULL NAME OF INVENTOR RESIDENCE &	DRAHOS CITY 147 00 Praha 4 POST OFFICE ADDRESS Na zlatnici 22 FAMILY NAME VOLF CITY		JIH STATE OR E CZECH CITY 147 00 MIRST GIVE VÁCIAV STAVE OR F CZECH	Praha 4 NAME OREIGN COUNTRY OREIGN COUNTRY	222	COUNTRY OF CZECH I	CMZENSHIP REPUBLIC CODE/COUNTRY REPUBLIC CM NAME CM IZENSKIP REPUBLIC CODE/COUNTRY
0. 2 2 2 5 6 6 6 7	OF INVENTOR RESIDENCE & CITIZENSHIP POST OFFICE ADDRESS FULL NAME OF INVENTOR RESIDENCE & CITIZENSHIP POST OFFICE ADDRESS reby declare that all st cure; and further this cor imprisonment, or dily of the application	DRAHOS CITY 147 00 Praha 4 POST OFFICE ADDRESS Na Ziatnici 22 FAMILY NAME VOLF CITY \$30 02 Pardubice POST OFFICE ADDRESS Devotyho 179 Interments made herein of my at these statements were made both, under section 1001 of it or any patent issuing there	y own knowledge ide with the kno Title 18 of the L	STATE OR ECZECH CITY 147 00 HIRST GIVE VÁCIAV STAYE OR ECZECH CITY 530 02 e are true an wledge that United State	Praha 4 NNAME OREIGN COUNTRY I REPUBLIC Pardubice d that all statements willful false statemes s Code, and that such	made on info rats and the willful false	COUNTRY OF CZECH I STATE & ZIP CI CZECH I STATE & ZIP CI CZECH I STATE & ZIP CI CZECH I Dination and like so made is statements i	CMZENSHIP REPUBLIC CODE/COUNTRY REPUBLIC CON NAME CONIZENSKIP REPUBLIC ODE/COUNTRY REPUBLIC belief are believ
0. 2 2 2 5 6 6 6 7	OF INVENTOR RESIDENCE & CITIZENSHIP POST OFFICE ADDRESS FULL NAME OF INVENTOR RESIDENCE & CITIZENSHIP POST OFFICE ADDRESS reby declare that all six of true; and further thing the continuous co	DRAHOS CITY 147 00 Praha 4 POST OFFICE ADDRESS Na Ziatnici 22 FAMILY NAME VOLF CITY \$30 02 Pardubice POST OFFICE ADDRESS Devotyho 179 Interments made herein of my at these statements were made both, under section 1001 of it or any patent issuing there	y own knowledgede with the kno	STATE OR ECZECH CITY 147 00 HIRST GIVE VÁCIAV STAYE OR ECZECH CITY 530 02 e are true an wledge that United State	Praha 4 NNAME OREIGN COUNTRY I REPUBLIC Pardubice d that all statements willful false statemes s Code, and that such	made on info	COUNTRY OF CZECH I STATE & ZIP CI CZECH I STATE & ZIP CI CZECH I STATE & ZIP CI CZECH I Dination and like so made is statements i	CMZENSHIP REPUBLIC CODE/COUNTRY REPUBLIC CON NAME CONIZENSHIP REPUBLIC ODE/COUNTRY REPUBLIC belief are believe

2	OF INVENTUR	VOZÁB		JAROSLAV		
0	residence a Cmzenchip	533 42 Živanice 39)	STATE OR FOREIGN COUNTRY CZECH REPUBLIC	CZX	COUNTRY OF CITIZENSHIP CZECH REPUBLIC
4	POST OFFICE ADDRESS	POST OPFICE ADDRESS 533 42 Živanice 3		orry 533 42 Živanice 39		STATE & ZIP OUDBECOUNTRY CZECH REPUBLIC
				4-44-4-4		
L						
					<u> </u>	
	<u> </u>					
			A CONTRACTOR OF THE CONTRACTOR			
	and belief a	re believed to be true, and and the like so made are p as Code, and that such wi	l further that those str junishable by fine or i	n knowledge are true and the atements were made with the imprisonment, or both, und may jeopardize the validity	he knowledge tha let section 1001 o	of Title 18 of the
sto	NATURE OF INVENTOR 20		SIONATURE OF INVENTO	R 203	signature of in	PLENTOR 206
DA1	18/1	12002	OA'IE		DAM	

PTO 1391 (REV. 1083) R/CCEPFPR/HERF-HAJEK-EU-1-PCT-COM-DEG-9 INV.-wpd Page 3 of 3

U.S. DEPARTMENT OF COMMERCE Fatons and Tradamark Office